

Original Article

Green synthesis of CuO micro-flowers using Hibiscus flower extract and study of photocatalytic degradation of water pollutant

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Manuscript ID: **Abstract**

JRD -2025(I)-170914

ISSN: 2230-9578

Volume 17

Issue 9(III)

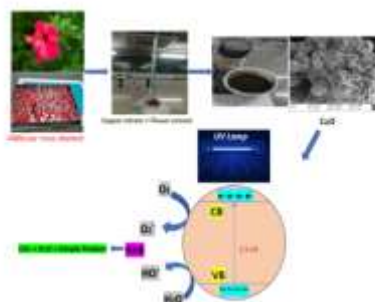
Pp. 79-86

Sept. 2025

Copper oxide micro flowers were effectively produced by the green approach in Hibiscus rosa-sinensis flower extract by co precipitation technique under mild reaction conditions. The produced materials were characterized for structural, morphological, and optical investigations using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and Ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis-DRS). The observed direct allowable band gap energy of synthesized CuO material is observed in the range of 3.5eV. SEM analysis demonstrates the creation of CuO micro flower crystallite material under mild reaction conditions using eco-friendly routes. Under the UV light irradiation, the synthesized CuO micro flower material efficiently degrades Rhodamine B dye solution up to 80% within 120min of irradiation. It has stabilized degradation ability against water polluting dye which has been monitored for two successful catalytic cycles.

Keywords: Green synthesis; Copper oxide microflower; Photodegradation; Optical band gap

Graphical Abstract:



Submitted: 12 Aug. 2025

Revised: 22 Aug. 2025

Accepted: 20 Sept. 2025

Published: 30 Sept. 2025

Introduction:

The contamination of the environment has significantly increased in the modern industrial era, especially in water caused by organic dyes, pesticides, and other harmful Chemical methods like Fenton, deformation, and adsorption have received a lot of attention lately for the decolorization of water contaminants by transition metal oxide catalysts because of their low toxigenicity, ideally with CO₂ and H₂O as the end products[1]. The energy source for several of these compounds was claimed to be artificial UV radiation. Nonetheless, UV in natural sunlight only makes up 5%–8% of the solar spectrum at sea level, which places a restriction on the catalysts and necessitates artificial illumination.

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How to cite this article:

Vikas Sawant. (2025). Green synthesis of CuO micro-flowers using Hibiscus flower extract and study of photocatalytic degradation of water pollutant. Journal of Research & Development, 17(9(III)), 79-86



Quick Response Code:



Website:
<https://jrdrv.org/>



The slow reaction rate to decolorize effluents, complex chemistry, and metal ion sludge are some of the additional drawbacks that make dye wastewater treatment difficult in practice.[2] Few studies have been published that examine CuO NPs' combined photocatalytic and antibacterial properties[2]. In these respects, because size influences the catalytic activity, it is essential to use a simple and cost-effective technique for creating CuO NPs with small diameters and slim sized distribution[3]. Although there are clear benefits, the expense, difficulty of use, and high power and energy requirements provide a significant disadvantage to the physical technique of synthesizing CuO NPs. Previously, floral extracts were used to create CuO NPs. Since the very beginnings of the development of numerous materials science domains, including optoelectronics and photonics, the scientific community has been interested in the functionalization of metal oxide nanoparticles through silver doping[4]. The application of silver doping has expanded to biomaterials, solar technologies, plasmonic, catalysis and sensoristics[5]. When it comes to photolytic, mechanical, and optical applications, CuO nanoparticles (NPs) respond appropriately. CuO NPs are created using a variety of techniques, such as sol-gel, solvothermal, microwave irradiation, hydrothermal, arc discharge, etc.[6]. It is believed that using plant extracts to produce CuO NPs is the most practical and eco-friendly approach. One of the many biological methods for producing CuO NPs is by using leaves, flowers, or stem extracts from plants like *Camellia japonica*[7], *Pterospermum acerifolium*[8], *Gum karaya*[9], and others as bioactive materials. An alternative to resolving the problems associated with the previously described approaches is the green synthesis technique, which promotes environmentally friendly chemistry by using plant extracts as reducing, stabilizing, and capping agents in the production of nanoparticles. With the availability of plant materials for large-scale nanoparticle manufacturing, the green synthesis process is economical, quick, sustainable, and environmentally benign. The Plant extracts have been used to generate CuO NPs in the past. The production of CuO NPs employed *Catha edulis* leaf extracts as reducing and capping agents. Another study produced CuO NPs for adsorption experiments using leaf extracts from *Eucalyptus Globulus*[10]. *Celastrus paniculatus* was also used to create CuO NPs using the secondary metabolites present in its leaf extracts. Leaf extracts were utilised in another study to create CuO NPs[11]. CuO NPs can be produced quickly, easily, affordably, and without harming the environment from plant extracts, as has been shown. A variety of metabolic and physiochemical mechanisms in plants depend on copper[12].

The textile, paper, and pharmaceutical sectors all utilize rhodamine B (RhB) dyes widely, although they are dangerous for the environment and human health. RhB is especially dangerous, poisonous, and may cause cancer. It can also linger in water, causing pollution and endangering aquatic life[13]. The Rhodamine family of dyes is significant. RhB and Rh 6G are the most widely used members of the Rhodamine family in a wide range of applications due to their higher chemical stability[14,15]. RhB-containing water has been treated using methods like photocatalytic degradation, ozonation, electrochemical approach, Fenton procedure, and others. Nowadays, photocatalysis is favoured over alternative dye degradation techniques because light irradiation on semiconductors produces electron and hole pairs that are utilized in the oxidation and reduction process[16]. The Respiratory infections, cancer, and neurotoxicity have all been linked to RhB because of its inherent structural stability and non-destructible nature[17]. A wide range of scientific and industrial fields make extensive use of metal nanoparticles (NPs) because of their remarkable optical, electrical, biological, and catalytic properties. The produced particles' size, shape, and chemical makeup greatly affect these characteristics[18]. For a number of metabolic and physicochemical functions, plants require copper. It is among the most important trace elements for the growth of plants[19]. Hibiscus flower extracts were used as capping and reducing agents in the synthesis of CuO NPs. The hibiscus extract itself is acidic, and its colour is dependent on the pH. It contains anthocyanins, the same pigment that is present in red cabbage and many other components of red plants. At low pH values, the extract will be green, and at higher pH values, it will be blue. Using several antioxidant systems, the flavonoid components of *Hibiscus rosa sinensis* petals were examined; the findings showed that the C5 compound of (*H. sabdariffa* var. *sabdariffa* race: *albus*) Commonly referred to as Indian sorrel, *Hibiscus sabdariffa* L. belongs to the *Malvaceae* family. It is widely grown in Mexico, India, and Africa for its edible components and berries, as well as for traditional medicinal purposes. Extracts from the plant's calyx and leaves have shown blood pressure-lowering benefits as well as anti-tumor, anti-cancer, hypotensive, choleric, diuretic, and chemo-protective qualities[20]. Using flower extract from *H. subdariffa*, created ZnO NP by environmentally friendly methods during work, schimat paying particular attention to how NP grows at various temperatures[21]. Thus, it can be assumed that hibiscus flower extract could produce nanoparticles stabilized by phytochemicals. We synthesized CuO micro flower polycrystalline materials in *Hibiscus* Flower extract in aqueous medium. The synthesized materials were characterized with spectrometric analysis and optical studies. The material has applications in efficient photodegradation of organic dye water pollutants such as Rhodamine B under UV light irradiation.

Experimental

1 Material and Methods:

For the preparation of copper oxide material; Pure AR grade Copper nitrate ($\text{Cu}(\text{NO}_3)_2$) was purchased from SD Fine chemicals, Mumbai as a precursor. Hibiscus flowers were collected from local area of Maharashtra. Double distilled water was used for preparation of plant extract and chemicals. The CuO microcrystallites were synthesized by ecofriendly rout at room temperature in laboratory. The prepared materials were characterized with double beam UV-

Vis-NIR spectrophotometer (Equiptronics; range 200nm – 1100nm); FTIR (FT/IR 4600 type A) Shimadzu; SEM (Zeiss, EHT=15kV);

2 Preparation of extract:

Hibiscus rosa-sinensis flowers were collected from local regions and were washed several times with water to remove the dust particle. The petals from flower were removed and collected for further process. It was dried in sunlight for 1hr and further dried in Tray dryer in laboratory (At 60°C for 2 hrs). The dried petals were cut in small pieces and grinded in mortar pestle to make fine powder. The 5gm of dried powder were placed in 250 ml glass beaker along with 100ml distilled water. The mixture was then boiled for 60 min until the colour of the aqueous solution changes from watery to dark pink colour by using magnetic stirrer. The extract was cooled to room temperature and filtered by using Whatman filter paper[22]. The extract was stored in refrigerator at below 10°C in order to be used for further experiment.

3 Preparation of CuO material:

The process of creating micro crystallite involved dissolving 10 gm of copper nitrate in 200 mL of distilled water, adding 100 mL of plant extract, and boiling the entire mixture for one hour in a water bath. This solution was boiled and then microwave for one hour at 250°C. After being collected in a ceramic crucible, this mixture was heated for two hours at 400°C in an air-heated furnace. For the purpose of characterization, a black powder was obtained and neatly packed. For characterization, the material was ground into a finer consistency using a mortar and pestle[23][24].

Result and Discussion :-

1 X ray diffraction analysis

The phase composition and crystallite structure of the synthesized CuO microcrystalline were determined with the aid of x-ray powder diffraction analysis. The Fig 1. shows the XRD pattern of synthesized CuO microcrystalline for the 2θ values in the range of 2.0 Degree to 8.0 Degree[25]. XRD was employed to study and explore the crystalline nature of the CuO microstructure material[23]. The average grain size of the material was obtained using the Debye-Scherrer's formula:

$$D = k\lambda / \beta \cos\theta$$

where "D" is the crystallite size (nm), "k" is Scherrer's constant, equal to 0.94, $\lambda = 1.54184 \text{ \AA}$ is the wavelength "β" is full width at half maximum (FWHM), and "θ" is the angle of diffraction.

The peaks are found at 2θ values of 32.4759, 35.4615, 38.7067, 48.7019, 53.5048, 58.3076, 61.5528, 66.2259, and 67.9134, which respectively, correspond to the crystal plane of 110, -111(002), (-200) -202, 020,202, -113,113, 220. The 2θ values and the crystal planes closely matched the JCPDS card number 48-1548 and 45-0937. The XRD pattern shown that the produced CuO microcrystalline were crystalline and represented the monoclinic tenorite phase of the CuO structure with calculated crystalline size 200nm shown in Table 1.

2 UV visible spectroscopy –

Synthesized CuO microcrystallites were analyzed by UV-Vis spectrum, which ranges from 200 to 800 nm, is shown in this Fig.2 For CuO microcrystallites, it shows the anticipated absorption band at 270 nm[26]. Stable CuO microcrystallites are synthesized in part by biocomponents present in plant extracts. A number of factors, including temperature, reaction time, precursor and flower extract concentrations, and microcrystallites shape, affect where the absorption band is located in the UV-Vis spectrum. There is a large concentration of microcrystallites in the sharp band. λ_{max} (Standard value) from literature = 262-280 nm and λ_{max} (Observed value) = 270 nm

3 Fourier Transform Infrared (FTIR) –

FTIR spectrum indicated the composition and functional groups present in Green synthesized CuO microcrystallites, scanned in the range from 600 to 4000 cm^{-1} . The intense bands of C-O, C-H, O-H and C-N found in the FTIR spectrum of CuO microcrystallites due to the involvement of Phytochemicals in Green Synthesized method found due to *Hibiscus plant* extract, which capped the CuO material[27]. Fig. 3 shows a broad band at 3456.78 cm^{-1} , which matches the stretching (O-H) hydroxyl functional group of water content found on the microcrystallites surface and at 16500 cm^{-1} is of (O-H) bending. The bands obtained at 2390 cm^{-1} nitrile group C-N stretching bond. The band at 1382.71 cm^{-1} can be ascribed to frequencies of the (C-H) group of carbon compound or biomolecules from flower extract. The influential stretching band of (C-O) of the plant extract bio element alcoholic group is found at 981.59 cm^{-1} . And at 529 cm^{-1} is peak of (Cu-O) bond[28]. This suggests that the biological molecules could possibly perform dual functional of formation and stabilization of Copper Oxide microcrystallites in the aqueous medium[29]. FTIR spectrum of CuO microcrystallites suggested that CuO microcrystallites were surrounded by different organic molecule.

4 SEM (Scanning Electron Microscopy) –

SEM (Scanning Electron Microscopy) technique is used to analyse synthesized CuO microcrystallites[30]. SEM micrograph occurs polycrystallites of CuO for the morphological study. The SEM image of Synthesized CuO material shows formation of microflowers as appear. The formation of polycrystalline material shown in Fig.4 (a) and (b) at low magnification (1μm) and high magnification(300nm). According to the SEM images which shows crystallites size between 200 nm to 300 nm which also concluded with the XRD data.

5. Optical properties of Material synthesis

The optical absorption analysis for material synthesis were carried out with UV-Vis-NIR double beam spectrophotometer using the data obtained from the optical absorption spectra. It has been shown the absorption maxima in UV and visible region. The CuO material synthesized by green synthesis method has absorption maxima in UV region. The synthesized CuO materials has ability to absorb UV light. The optical energy band gap, E_g of CuO material synthesis were estimated by plot of $(\alpha h\nu)^2$ against the photon energy ($h\nu$). In order to get the value of the direct allowed optical band gap (E_g), extra plotting the straight line to intercept photon energy axis which gives values of band gap energy (E_g) as in the range of 3.52eV shown in Fig.5

Application

1 Photocatalytic degradation of Rhodamine-B dye

The photo catalytic activity of synthesized CuO material were studied against Rhodamine-B dye under Ultra violet light. For that 100mg of as synthesized CuO material was taken in 100 ml of 5 ppm Rhodamine-B dye solution in 250 ml reaction beaker. Before irradiation to UV light the solution was kept in the dark for at least 1 hr allowing the adsorption/desorption equilibrium to be reached. After that the solution was kept under UV light. First sample was taken before the irradiation to obtain dark adsorption, which was after considered as the initial concentration ($A_{initial}$). Samples were then withdrawn regularly from the reaction beaker by an order of 30min, 60 min, 90 min, 120 min. The sample of 120min was taken as final (A_{final}). The solution was then analysed by using a UV-Vis-NIR spectrophotometer at wavelength range from 400 to 800 nm. The degradation of Rhodamine B under UV light was monitored for two successive catalytic cycles as shown in Fig 6. Cycle (a) and (b). It has been observed that the CuO catalyst has 80% of stability for degradation of Rhodamine B. The efficiency of material was calculated by using following equation.

$$\text{Efficiency}(\%) = \frac{A_{initial} - A_{final}}{A_{initial}} \times 100 \dots\dots\dots 2$$

It was observed that CuO material synthesized by Green-synthesis method has 80% efficiency for degrades Rhodamine-B dye solution [31] shown in Fig.7. The free electrons probably react with dissolved oxygen molecules and produce oxygen peroxide radical $O_2^{\cdot-}$ the positive charged hole (h^+) may react with the OH^- derived from H_2O to form hydroxyl radical $OH^{\cdot}$. The Rhodamine-B molecule then can be photo catalytically degraded by oxygen peroxide radical $O_2^{\cdot-}$ and hydroxyl radical $OH^{\cdot}$ to forms CO_2 , H_2O and other mineralization products (P). The reaction kinetics was studied by plotting of absorption intensity (a.u.) against Time (min) for the degradation of Rhodamine-B solution using CuO material as shown in Fig.6 cycle (a) and (b). [2]

Conclusion:

- CuO micro flower crystallites were successfully synthesized by green rout using *Hibiscus* Flower extract in aqueous solution.
- Through UV-Vis-NIR absorption, the optical characteristics of CuO nanoparticles were examined and clarified and the band gap range is 3.5eV and Absorption maxima at 270nm.
- The prepared CuO material has crystalline nature and represented the monoclinic tenorite phase with crystallite size of 200nm.
- The synthesized CuO material has 80% of efficiency for degradation of Rhodamine B dye solution under UV light and has high catalytic stability for two successive cycles.

Acknowledgements

The authors would like to acknowledge the CFTIR Institute Mysore, India for providing SEM analysis facility. The authors are also grateful to The Head, D Y Patil Agriculture and Technical University, Talsande for providing Laboratory facilities during the research work.

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Figures

Figure Captions:

Figure.1 XRD Pattern of CuO Micro crystallite

Figure.2 UV-Vis Spectrum of CuO Micro crystallite

Figure.3 FTIR Spectra for CuO

Figure.4 SEM Images of CuO Micro crystallite

a) at Low magnification b) at High magnification

Figure.5 Graph of $(\alpha h\nu)^2$ against the photon energy ($h\nu$)

Figure.6 UV absorption graph for Photodegradation of Rhodamine B

Figure.7 The graph of Intensity Vs time for calculating efficiency

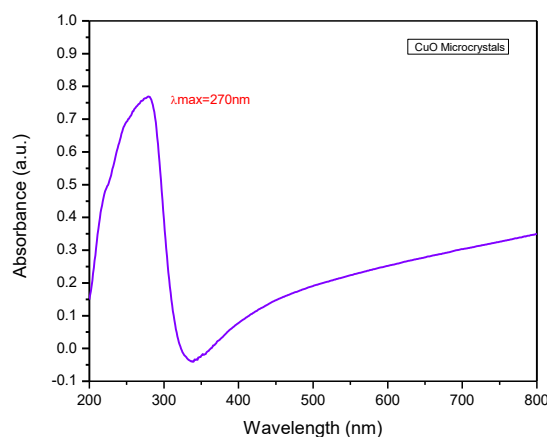


Fig.2

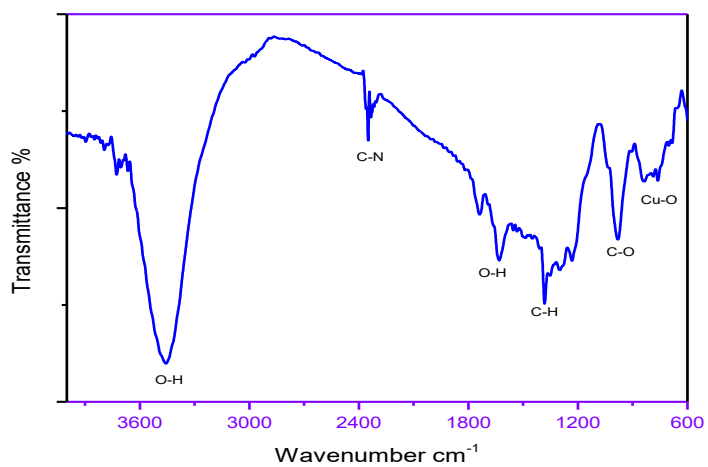
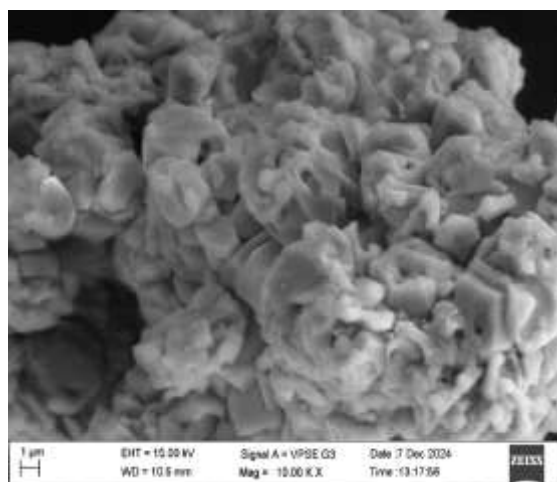
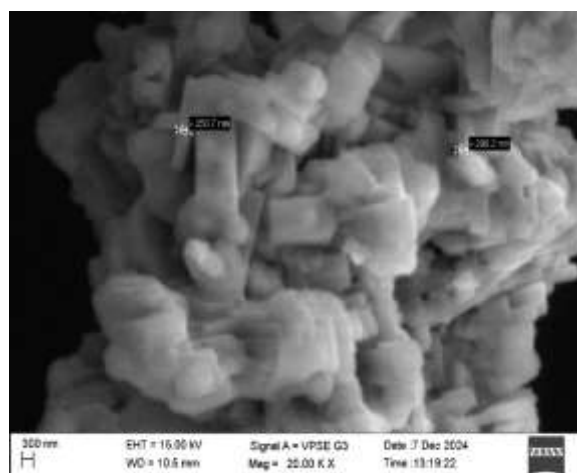


Fig.3



(a)



(b)

Fig.4

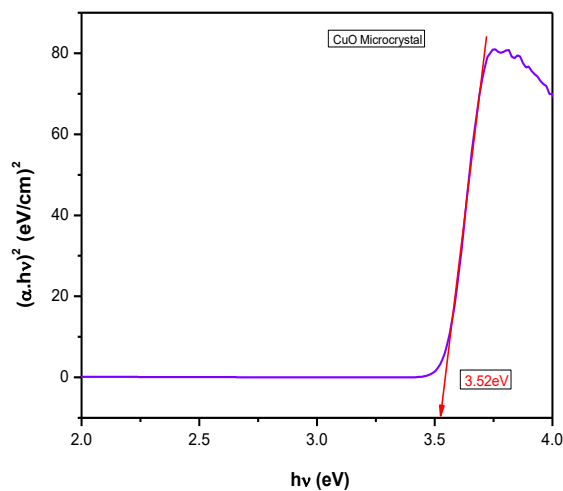
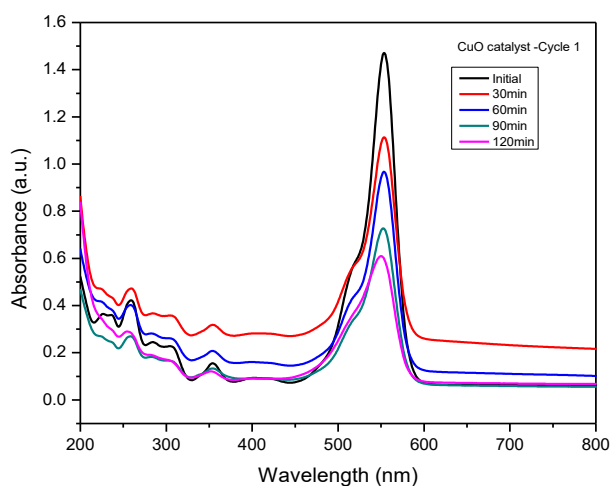
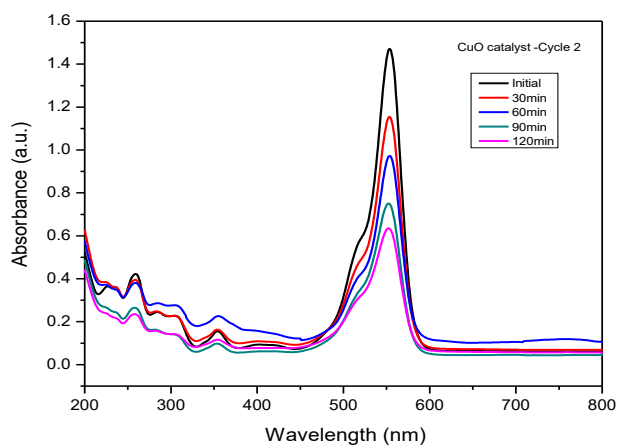


Fig.5



Cycle (a)



Cycle (b)

Fig.6

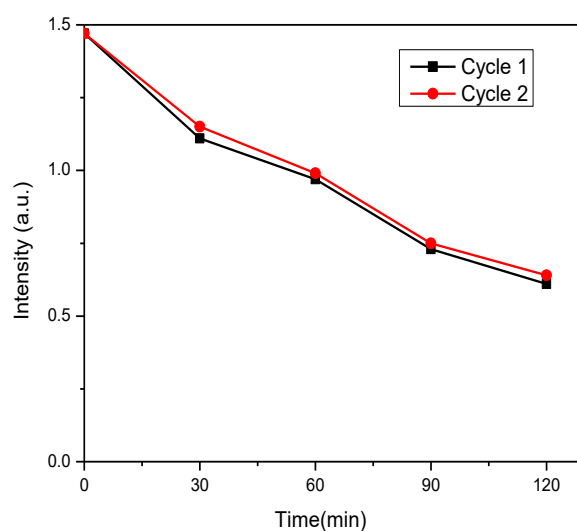


Fig.7

Table.1 Crystallographic parameters of the synthesized CuO crystallites based on the X-Ray diffraction pattern.

2θ values		hkl Planes	d values (Å ⁰)	Crystalline size (nm)
Standard (JCPDS #48-1548)	Observed			
32.6	32.4759	110	2.84	200 nm
35.7	35.4615	-111(002)	2.62	
38.3	38.7067	(-200)	2.31	
49.0	48.7019	-202	1.93	
53.6	53.5048	020	1.75	
49.0	58.3076	202	1.62	
61.7	61.5528	-113	1.57	
66.2	66.2259	113	1.46	
68.4	67.9134	220	1.42	